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(54) Production method of D-glucosamine hydrochloride

The invention relates to the method of production of D-glucosamine hydrochloride (2-amino-2-deoxy-D-glucose hydrochloride).

What is known is the production of D-glucosamine hydrochloride from natural materials containing chitin, e.g., shells of crustaceans or chitin organs of other arthropods (M. Stacey & J. M. Weber, Method Carbohydr. Chem. 1, 228-230 (1962)).

The main disadvantage of the original production method using crab shell chitin is the unavailability of crab in our environment and that is why this raw material must be imported.

The stated insufficiencies are eliminated according to the disclosed production method for D-glucosamine hydrochloride. It is characterized by the fact that structural polysaccharide from the mycelia of the imperfect fungus *Aspergillus niger* can be obtained by the effect of suitable alkali solutions. For example, mycelium can be hydrolyzed at a temperature of 80 to 100°C in hydrochloric acid. After the hydrolysis, excess hydrochloric acid can be removed through evaporation and thickening. Then, after ethanol is added, the glucosamine hydrochloride is left to crystallize at room temperature. The benefit of the new method is the easy availability of the raw material. The mycelia of the imperfect fungus *Aspergillus niger*, a byproduct of citric acid production, can be used to produce chitin.

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The production method, according to the invention, is further specified by practical examples.

Example 1

680 g of moist mycelium collected after completed fermentation during the production of citric acid, containing 80% dry matter, was suspended in 700 ml of 10% NaOH and heated in a boiling hot bath for 2 hours and then for another 2 hours on an electric stove.

The suspension of raw polysaccharide was separated by centrifuge and sediment was suspended three times and rinsed with water, centrifuged, and suspended in 900 ml of 50% citric acid. It was partially decolorized by adding solid sodium sulfite (four times 2 g). It was rinsed with water, rinsed with ethanol three times, and dried at room temperature. Gain: 37 g of polysaccharide.

Example 2

Dry polysaccharide (35 g) was suspended in 170 ml of concentrated HCl and the mixture was heated in a boiling water bath for 2.5 hrs. The same volume of water was added after this and the suspension was filtered through diatomite. Activated charcoal was added to the brown filtrate and the suspension was again filtered through diatomite.

Light yellow solution was evaporated and thickened at 60°C under

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reduced pressure to 30 ml. 120 ml of ethanol was added to this solution and the mixture was left to crystallize for 24 hours at laboratory temperature. The eliminated product was filtered, gradually washed by ethanol and ether, and dried out in the open.

Gain: 8.5 g.

The α -anomer crystallizes following re-crystallization from the smallest amount of hot water and four volumes of ethanol and β -anomer is left in the solution, which can be gained through ether precipitation. α -Anomer forms colorless needle-like crystals with a decomposition point of 190–194°C and specific optical rotary power $[\alpha]_D^{25}$

+ 100 $\rightarrow$ +72°, (c 1.0, H₂O, tube length 10 cm).

Example 3

Polysaccharide suspension with 680 g of moist mycelium was collected after de-colorization by sodium sulfite and rinsed, suspended in 170 ml of concentrated HCl, according to the method stated in Example 1, and centrifuged and processed, as stated in Example 2.

Gain: 8.5 g of product with similar properties as stated in Example 2.

SUBJECT OF INVENTION

Method of D-glucosamine hydrochloride production characterized by the fact that the structural polysaccharide from the mycelium of the imperfect fungus *Aspergillus niger*, is collected by the effects of water solutions of alkali, e.g., also sodium hydroxide, on mycelium at a temperature of 80 to

100°C, is hydrolyzed by citric acid, whereas the hydrolysis removes excess hydrochloric acid and, following the addition of ethanol, the D-glucosamine hydrochloride is left to crystallize at room temperature.



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from Czech into English.


Jinmin Lee

Sworn to before me this
4th day of June, 2009


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